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Improved Electrolytes for Lithium Rechargeable Batteries

Charles W. Walker, Jr.

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13. ABSTRACT (Maximum 200 words) There has been a continuing effort to identify and develop chemistries that will satisfy the stringent MILSPEC requirements for a rechargeable lithium battery. Cell potentials exceeding 4.0 V present a difficult task to find electrolytes which are conductive and stable to oxidation at high potentials. In this study we selected four lithium salts and three solvents from which electrolytes were prepared and evaluated with respect to conductivity, electrochemical stability, and stability with aluminum (an issue because of its use as a current collector at the cathode). We have identified a conductive electrochemically stable electrolyte comprised of lithium tris-(trifluoromethanesulfonyl)methide in a ternary solvent mixture of ethylene carbonate, dimethyl carbonate, and methyl formate as a viable candidate for a military electrolyte. We have also shown that a salt believed unstable with aluminum, lithium bis-(trifluoromethanesulfonyl)imide, is stable with aluminum, and is another candidate for a suitable electrolyte.				
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Introduction

One of the most important factors involved in the development of a commercially successful rechargeable lithium battery has been to identify a suitable electrolyte. Whether utilizing metallic lithium or a lithium ion intercalating anode, there is a continuous effort to develop improved electrolytes which are conductive, chemically and electrochemically stable, exhibit good cycling efficiency, and are safe. Because military specifications (e.g., operating temperature range from -30°C to 55°C) are more extreme than commercial requirements, this task becomes even more difficult.

The currently preferred rechargeable lithium battery chemistry is based on a lithium intercalating carbon anode (e.g., graphite, petroleum coke), a lithium intercalating metal oxide cathode (e.g., LiNiO_2 , LiCoO_2), and an electrolyte composed of a lithium salt dissolved in a mixture of at least two organic solvents. In fact, there are commercially available batteries with this general composition. Even so, improvements are desired in both the electrolyte and the electrode compositions.

Electrolytes containing LiAsF_6 are known to be highly conductive even at low temperature and are able to support high rate discharges. Even so, the use of LiAsF_6 raises issues of toxicity and carcinogenicity which make this a reluctant choice for a commercial product. Presently available commercial batteries contain LiPF_6 , but there have been questions raised about its stability.

We began this study with the assumption that both LiAsF_6 and LiPF_6 will be undesirable electrolyte components. On this basis, we began screening electrolytes containing two other lithium salts, $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ and $\text{LiC}(\text{CF}_3\text{SO}_2)_3$, in ethylene carbonate:dimethyl carbonate (EC:DMC) and EC:DMC:Methyl Formate (EC:DMC:MF) solvent mixtures. Tests included conductivity of electrolyte solutions over the MILSPEC temperature range and electrochemical stability using glassy carbon, platinum and aluminum substrates.

Experimental

Electrolytes were prepared with high purity salts that were dried overnight under vacuum at 100° - 115°C . EC (Aldrich, 98%) was dried and stored over 4Å molecular sieves. DMC and MF (Aldrich) were distilled over a stream of dry argon and stored over 4Å molecular sieves. Salt concentrations were fixed at one molar (no salt mixtures were explored at this time). Binary and ternary solvent systems were prepared in volume ratios of 1:1 and 1:1:1 respectively. No attempts were made to optimize salt concentration or solvent ratios.

Solutions were prepared in a dry (<2 ppm H_2O) argon-filled glove box. All of the EC-DMC binary electrolytes were clear and appeared to be stable at least

over a period of several weeks. We know from the literature that LiAsF_6 is not stable with MF, but that stability increases in solvent mixtures as $\text{MF} \ll \text{DMC} < \text{MF/DMC} < \text{MF/DEC} < \text{DEC}$ [1]. A 1M LiPF_6 -EC-MF solution was prepared, but was not used because it turned bright orange, indicating some reaction between the salt and MF. We also observed reduced stability with the ternary MF-containing electrolyte, 1 M LiPF_6 -EC-DMC-MF. Initially this solution was clear, but over the course of several days a pale orange tint developed. The extent of this instability was not determined, but we proceeded with conductivity and electrochemical stability tests.

The 1 M $\text{LiC}(\text{CF}_3\text{SO}_2)_3$ -EC-DMC-MF electrolyte appeared to be stable as no color changes were observed. However, the solution was slightly cloudy as prepared. We suspect that the cloudiness may indicate 1 M lithium methide exceeds the solubility limit for this ternary solvent mixture.

Conductivities were performed in a sealed conductivity cell and resistance measured with a Wayne Kerr model 6425 bridge. Temperature was controlled in a Tenney box and measured with a Fluke digital thermocouple. The cell was thermally equilibrated at each temperature for approximately 1.5 hours or longer as deemed appropriate. To ensure that no changes were occurring in the electrolytes during course of taking measurements at all temperatures, we skipped temperatures as we decreased or increased cell temperature, filling in the "missing" temperature reading when returning to temperatures in the opposite direction. In no instances did any discrepancies arise, since the "in-between" values fell in line to form congruous sets of data.

Cyclic voltammetric sweeps were conducted in glass cells flooded with each electrolyte and containing a lithium counter electrode and Li/Li^+ reference electrode. Oxygen and any other dissolved gases were purged from the electrolytes by bubbling ultra high purity argon after first passing the gas through a bubbling tube containing dry DMC. The reason for the bubbler was to "moisturize" the dry argon (with relatively volatile DMC) to prevent selectively picking up or driving off solvents in the electrolyte. Three different working electrodes were used to evaluate stability of the electrolyte and compatibility with the electrode materials. We employed conventional glassy carbon and platinum working electrodes polished to a mirror finish with 0.1 μm alumina as well as a 99.999% pure polished aluminum working electrode to evaluate stability of the electrolytes with aluminum (since Al is the usual current collector for metal oxide cathodes). All sweeps were performed at a 5 mV s^{-1} scan rate via an EG&G PAR Model 273 potentiostat/galvanostat controlled by Model 270 software.

A Perkin Elmer TGS-2 Thermogravimetric Analyzer was used to measure decomposition temperature for determining thermal stability of LiPF_6 . Samples

were evaluated from 25°C to 300°C at a heating rate of 10°C/min over a slow flow of either dry argon or room air.

Discussion

Solvents. A suitable single solvent electrolyte has not been identified for rechargeable lithium systems, but unexpected synergism between two or more solvents has shown promise in the past [1,2]. From the literature, we narrowed our list of considered solvents to propylene carbonate (PC), ethylene carbonate (EC), dimethyl carbonate (DMC), and methyl formate (MF). Some physical constants are given in Table 1. PC appears to be an attractive solvent except for its high viscosity which results in poor conductivity at low temperatures, and that it is electrochemically reduced at the lithium electrode.

Table 1. Properties of Candidate Solvents

Solvent	mp/°C	bp/°C	ϵ	η/cP
PC	-55	241	64.4	2.54
EC	36.4	238	90-95	1.85
DMC	0.5	90	3.12	.585
MF	-99	31.5	8.9	.328

In MF, stability of LiAsF_6 is poor and cycle life is low [1]. Although LiAsF_6 is very stable in diethyl carbonate (DEC), viscosity is high and cycling efficiency is nearly 0%. However, a mixture of MF/DEC allows (unexpectedly) for a cycling efficiency of 83%. An MF/ CO_2 mixture which also contains some LiBF_4 in the electrolyte is 98% efficient [1]. MF/DMC also has shown good cycling efficiency. On this basis, only solvent mixtures were considered.

Eliminating PC, we decided to pursue only solvent mixtures containing EC-DMC and EC-DMC-MF, expecting to maintain good ionic conductivity while reducing viscosity and lowering the freezing point of the electrolyte.

Salts. The LiPF_6 and LiAsF_6 salts were used to provide baseline values with which to compare the two salts selected for study: lithium bis-(trifluoromethanesulfonyl)imide, $\text{LiN}(\text{CF}_3\text{SO}_2)_2$, and a newly synthesized salt, lithium tris-(trifluoromethanesulfonyl)methide, $\text{LiC}(\text{CF}_3\text{SO}_2)_3$. The three most important factors to be considered were thermal stability, electrochemical stability, and the ability to form conductive non-aqueous solutions.

Although it has been reported that LiPF_6 has poor thermal stability there was no information available from the manufacturer other than it decomposes to phosphorous oxides and a poisonous gas, phosphine. We felt it was necessary to first determine if the salt by itself or as a component of an electrolyte could withstand temperatures within the MILSPEC temperature range. We also

investigated temperatures above 55°C since higher temperatures are not uncommonly encountered in, for instance, a vehicle parked in the sun.

Initially, a simple visual test was conducted with 1M LiPF₆-EC:DMC (1:1) and 0.5 M LiN(CF₃SO₂)₂-EC:PC:DMC (1:1:3) electrolytes. The imide salt is known to be thermally stable, so it served as a control. Sealed test tubes containing electrolyte were placed in a sealed glass oven packed with dry molecular sieves to help maintain even heating. The tubes were placed against the oven wall so continuous observations were possible without removing the tubes from the oven. Temperature was raised in increments, observing for color changes in the electrolyte that indicate decomposition. Results are reported in Table 2.

Table 2. Thermal Stability of Electrolytes.

°C	Hrs	LiPF ₆	LiN(CF ₃ SO ₂) ₂
40.5	16	NC	NC
47.5	24	NC	NC
61.4	24	NC	NC
77.6	7	yellow	NC
	24	dark brown	NC

NC = no change

The imide electrolyte appeared stable at all temperatures (up to 24 hours). The LiPF₆ electrolyte was stable at 61.4°C, but quickly began turning color at 77.6°C, becoming dark brown within 24 hours. Therefore, this electrolyte decomposes at some temperature between 62°C and 78°C. From this observation, we confirmed that electrolyte containing LiPF₆ is not stable enough for military applications.

We then determined thermal stability of LiPF₆ salt by thermogravimetric analysis (TGA), whereby a small sample was exposed to continuously increasing temperature while weight loss was measured during decomposition. TGA was performed on dry LiPF₆ by two different methods: with a slight flow of dry argon and with room air atmosphere. Results were similar in both cases. There was a two step decomposition, with the onset of weight loss occurring at 68°C. A sharp knee ending at about 100°C was followed by a sloping plateau which accounted for a weight loss of approximately 12%. Another weight loss occurred continuously beginning at about 180°C to about 260°C, with an additional 61% weight loss for a total weight loss of 83.1%. No further weight loss occurred up through 300°C.

To satisfy ourselves that this low temperature weight loss was not water being lost and that our salt was truly dry, two additional experiments were run. "Wet" LiPF₆ salt was prepared by letting the salt stand open to the air for an

hour, with measurements performed under dry argon. "Wet" salt showed an immediate weight loss which continued to 300°C. In this case, only 64.2% of the total weight was lost. It is likely that moisture had caused the formation of thermally stable phosphorous oxides or other thermally stable compounds before the onset of the experiment. Next, we ran NaCl, dry and wet (drop of water added). Dry NaCl showed absolutely no weight loss to 300°C as expected (melting point is 801°C). The wet NaCl resulted in a profile similar to that seen with the wet LiPF₆, assuring us that the weight loss observed between 68°C and 105°C with LiPF₆ was really due to the onset of decomposition of the salt rather than loss of water.

In addition to dry LiPF₆ decomposing at 68°C, there are reports that trace amounts of HF or water can autocatalyze the decomposition of LiPF₆ at even lower temperatures in the 40°C range.

The first salt of interest, lithium bis-(trifluoromethanesulfonyl)imide, LiN(CF₃SO₂)₂, has been of great interest for both liquid and solid polymer electrolytes. The charge delocalization of the large anion serves to reduce ion pairing [3]. Conductivity is fairly good even though this salt increases electrolyte viscosity. Although thermally stable to 360°C, there has been some question about the stability of this salt with aluminum current collectors. Therefore, we were particularly interested in determining the electrochemical stability of this salt with aluminum of high purity (99.999%) to resolve this question.

The second salt of interest was a newly synthesized methide salt, lithium tris-(trifluoromethanesulfonyl) methide, LiC(CF₃SO₂)₃. As shown in Table 3, it exhibits both thermal stability and good room temperature conductivity [4,5]. This salt was recently patented by Covalent Associates, Inc. [4] but is not yet commercially available. Recent work shows the methide has better electrolyte conductance and anion stability than other lithium salts (LiClO₄, LiAsF₆, LiPF₆, LiN(CF₃SO₂)₂, LiCF₃SO₃) in both aprotic solvents and water [6].

Table 3. Properties of Candidate Lithium Salts.

Salt	mp (°C)	Decomp. Temp by TGA (°C)	$\sigma/S\text{ cm}^{-1}$ as 1M THF soln, 25°C
LiPF ₆	-----	68 or less	-----
LiAsF ₆	-----	(2 reports) 240, 350	-----
LiN(CF ₃ SO ₂) ₂	236-237	360	1×10^{-2}
LiC(CF ₃ SO ₂) ₃	271-273	340	1.2×10^{-2}

Electrolyte Conductivity. Conductivity as a function of temperature was performed in a sealed conductivity cell which was placed in a Tenney box to control temperature. Electrolyte conductivity between -30°C and 55°C is shown in Figures 1 and 2, and a few values are also given in Table 4. Note that for the binary solvent systems, those containing LiPF₆, LiAsF₆ and LiN(CF₃SO₂)₂ all become frozen before -30°C. Only the lithium methide binary electrolyte remains liquid at -30°C, which is clearly reflected in the conductivity (Figure 1). At room temperature and above, conductivity decreases as LiAsF₆ \approx LiPF₆ > LiN(CF₃SO₂)₂ > LiC(CF₃SO₂)₃.

The addition of MF in formulating the ternary electrolytes successfully prevented freezing at -30°C and significantly increased conductivity at all temperatures (Figure 2). Although conductivity with LiPF₆ is approximately 1.5 times greater than the lithium methide electrolyte, we know that only the methide is thermally stable. Above room temperature, the conductivity for the ternary lithium methide electrolyte is as good or better than all of the binary electrolytes. Below room temperature this electrolyte exhibits much better conductivity than all of the binary electrolytes, especially below -15°C. We expect that in the ternary solvent system the lithium imide salt would show superior conductivity.

Table 4. Electrolyte Conductivity and Freezing Temperatures.

Electrolyte	Freezing °C	Conductivity / S cm ⁻¹		
		-30 °C	25 °C	55 °C
1M LiAsF ₆ in EC:DMC	≥ -19.7	2.6 x 10 ⁻⁴	1.1 x 10 ⁻²	1.8 x 10 ⁻²
1M LiPF ₆ in EC:DMC	≥ -15.4	1.7 x 10 ⁻⁴	1.1 x 10 ⁻²	1.8 x 10 ⁻²
1M LiN(CF ₃ SO ₂) ₂ in EC:DMC	≥ -29.0	3.4 x 10 ⁻⁴	9.0 x 10 ⁻³	1.4 x 10 ⁻²
1M LiC(CF ₃ SO ₂) ₃ in EC:DMC	NO	1.1 x 10 ⁻³	7.1 x 10 ⁻³	1.1 x 10 ⁻²
1M LiPF ₆ in EC:MF:DMC	NO	5.4 x 10 ⁻³	1.8 x 10 ⁻²	2.5 x 10 ⁻²
1M LiC(CF ₃ SO ₂) ₃ in EC:MF:DMC	NO	3.5 x 10 ⁻³	1.2 x 10 ⁻²	1.8 x 10 ⁻²

Electrochemical Stability. Not only must an electrolyte exhibit acceptable ionic conductivity, but electrochemical stability must also be demonstrated. To determine stability, each electrolyte was evaluated with all three working electrodes for single and multiple sweeps to oxidation potentials as high as 4.3 V. Although identical data were collected for all four salts, the LiPF₆ and LiAsF₆ sweeps were run primarily for purposes establishing baselines of comparison. Therefore, the following discussion will mainly focus on the lithium imide and lithium methide electrolytes since these are the candidates most likely to satisfy the “military electrolyte” requirements.

Figure 3 shows a single sweep comparison for all of the binary solvent electrolytes with glassy carbon between 2.4 V and 4.1 V. Current density is low for all electrolytes and the general shape of the curves is approximately the same as well. Since good cell cycling behavior with the LiPF_6 electrolyte has been established in commercial batteries, and the voltammograms are similar for LiAsF_6 , $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ and $\text{LiC}(\text{CF}_3\text{SO}_2)_3$, we assume that all of the binary electrolytes will be stable within this "normal" range of potentials.

Figure 4 shows voltammograms for glassy carbon in two ternary electrolytes. The LiPF_6 electrolyte looks similar to the binary systems. The $\text{LiC}(\text{CF}_3\text{SO}_2)_3$ electrolyte shows an increased current density at the voltage limits from about $20 \mu\text{A cm}^{-2}$ to $35 \mu\text{A cm}^{-2}$, which is still very low.

In the following figures the oxidation potentials go to 4.3 V, which usually describes an overcharge (abuse) condition for most cathode materials. In Figure 5, all three types of working electrodes are shown with the $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ -EC-DMC electrolyte. Although there have been reports that aluminum is unstable with the imide salt, we found the aluminum electrode to be the most stable, showing very little current even under this "abuse" oxidation potential. The most active substrate is the platinum, which shows no more than $90 \mu\text{A cm}^{-2}$ at 4.3 V.

In Figure 6, all three types of working electrodes are shown with the $\text{LiC}(\text{CF}_3\text{SO}_2)_3$ -EC-DMC-MF ternary electrolyte. Again, the aluminum substrate is the least reactive, while platinum and glassy carbon show oxidation and reduction maxima of less than $70 \mu\text{A cm}^{-2}$.

In Figure 7 we show the comparison between three electrolytes, all conducted with an aluminum working electrode. Interestingly, the ternary methide electrolyte is the most stable while the binary methide system has the highest current response. However, it should be noted that the highest response is merely $25 \mu\text{A cm}^{-2}$, indicating exceptional stability with aluminum for all three electrolytes. These results assured us that stability with an aluminum current collector is not an issue with either the lithium imide salt or the lithium methide salt.

In Figure 8 we show multiple sweeps on aluminum for the $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ -EC-DMC and $\text{LiC}(\text{CF}_3\text{SO}_2)_3$ -EC-DMC-MF electrolytes. Sweeps one (upper curves) and five are plotted to indicate the effect of cycling on the current response with aluminum. In both electrolytes, the fifth cycle shows significantly less activity at the aluminum electrode. This is important and preferred since it demonstrates that the already small oxidation currents (at 4.3 V) are diminishing with each subsequent sweep due to passivation of the aluminum. This means that during cell cycling we can expect negligible currents due to oxidation of the

electrolyte on the aluminum current collector, further attesting to the stability of the aluminum current collector with these electrolytes.

Conclusions

In satisfying the requirement for a "military electrolyte" capable of performing between -30°C and 55°C we have identified two promising candidate salts which are thermally stable, electrochemically stable, and able to form ionically conductive solutions. A lithium methide ternary electrolyte, $\text{LiC}(\text{CF}_3\text{SO}_2)_3\text{-EC-DMC-MF}$, shows good conductivity at all temperatures and is stable with an aluminum substrate even to 4.3 V. The lithium imide salt, $\text{LiN}(\text{CF}_3\text{SO}_2)_2$, is also considered to be a viable candidate. Contrary to reports about it being incompatible with aluminum, we found this salt to be as stable as LiPF_6 , LiAsF_6 , and $\text{LiC}(\text{CF}_3\text{SO}_2)_3$. The imide salt in an EC:DMC binary electrolyte suffers from poor conductivity and freezing at low temperature, but this could probably be remedied by using a ternary EC:DMC:MF solvent system as demonstrated here with the LiPF_6 and $\text{LiC}(\text{CF}_3\text{SO}_2)_3$ salts. In addition, we would expect this combination to provide better conductivity than the methide ternary electrolyte.

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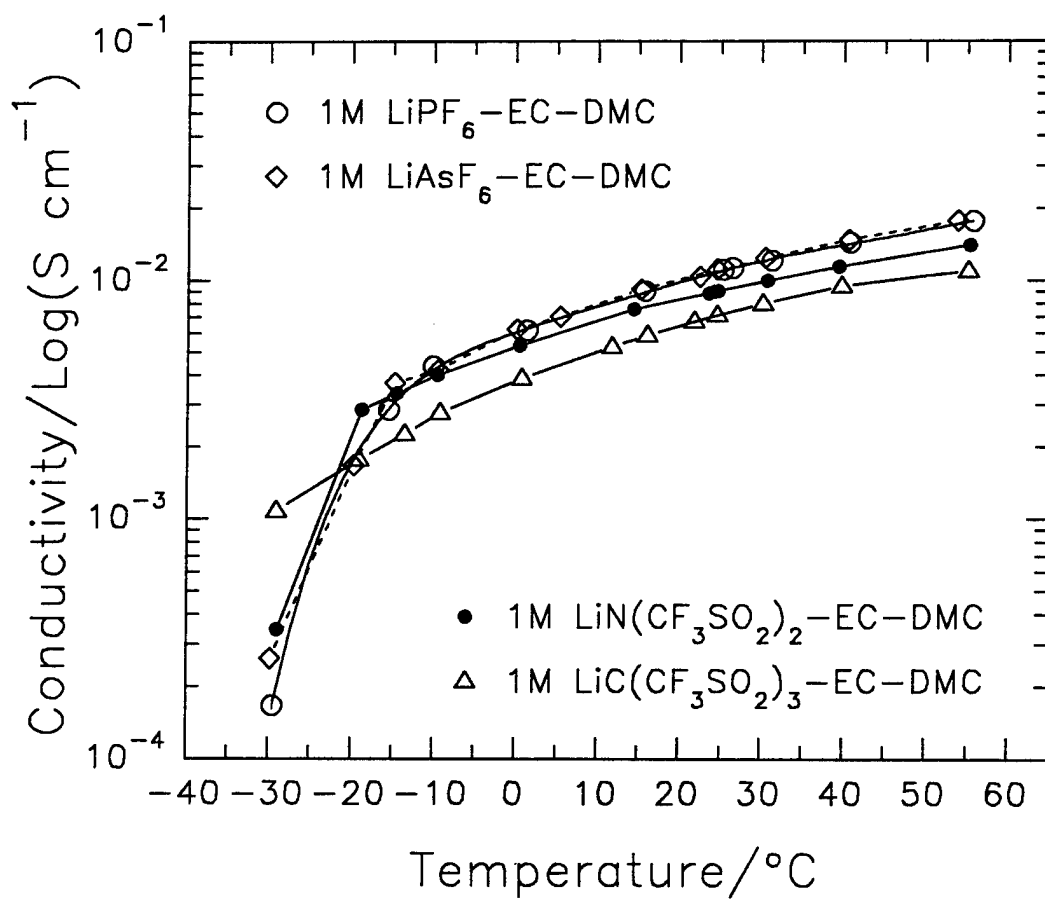


Figure 1. Electrolyte conductivity as a function of temperature for binary solvent electrolytes.

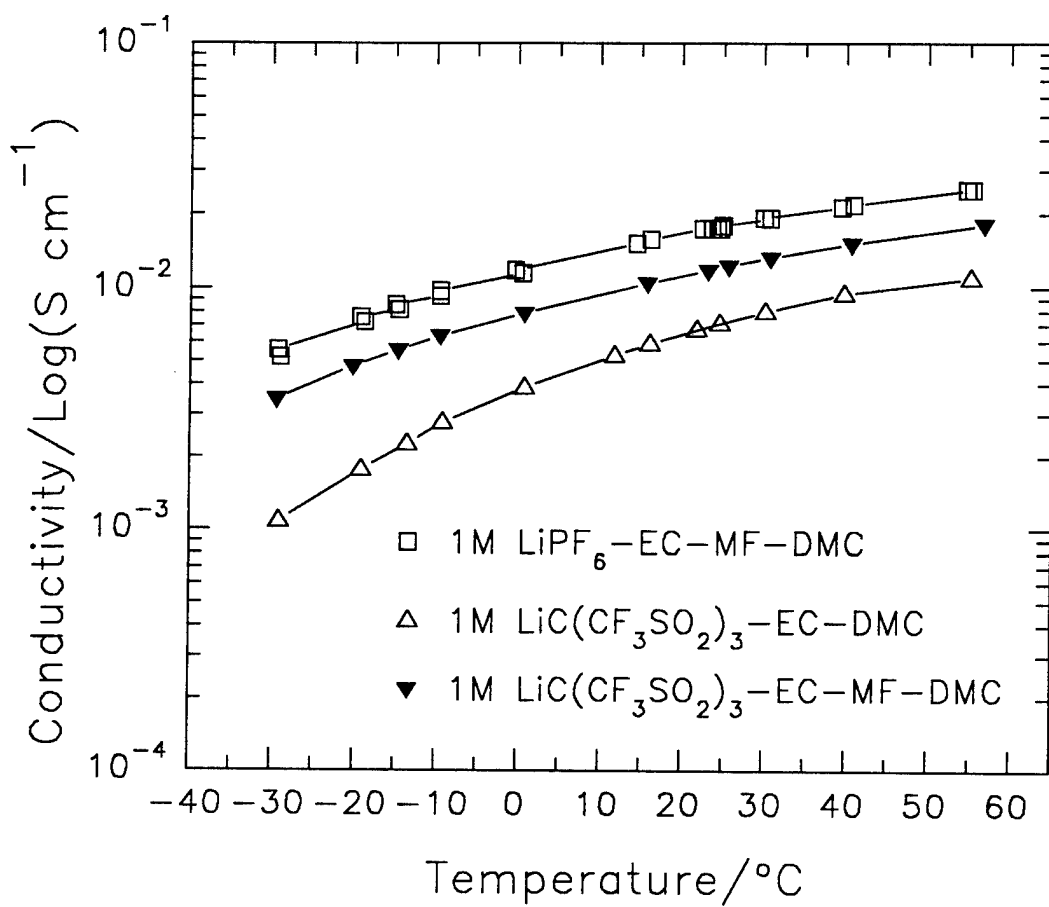


Figure 2. Electrolyte conductivity as a function of temperature for ternary solvent electrolytes compared to the binary electrolyte containing lithium methide salt.

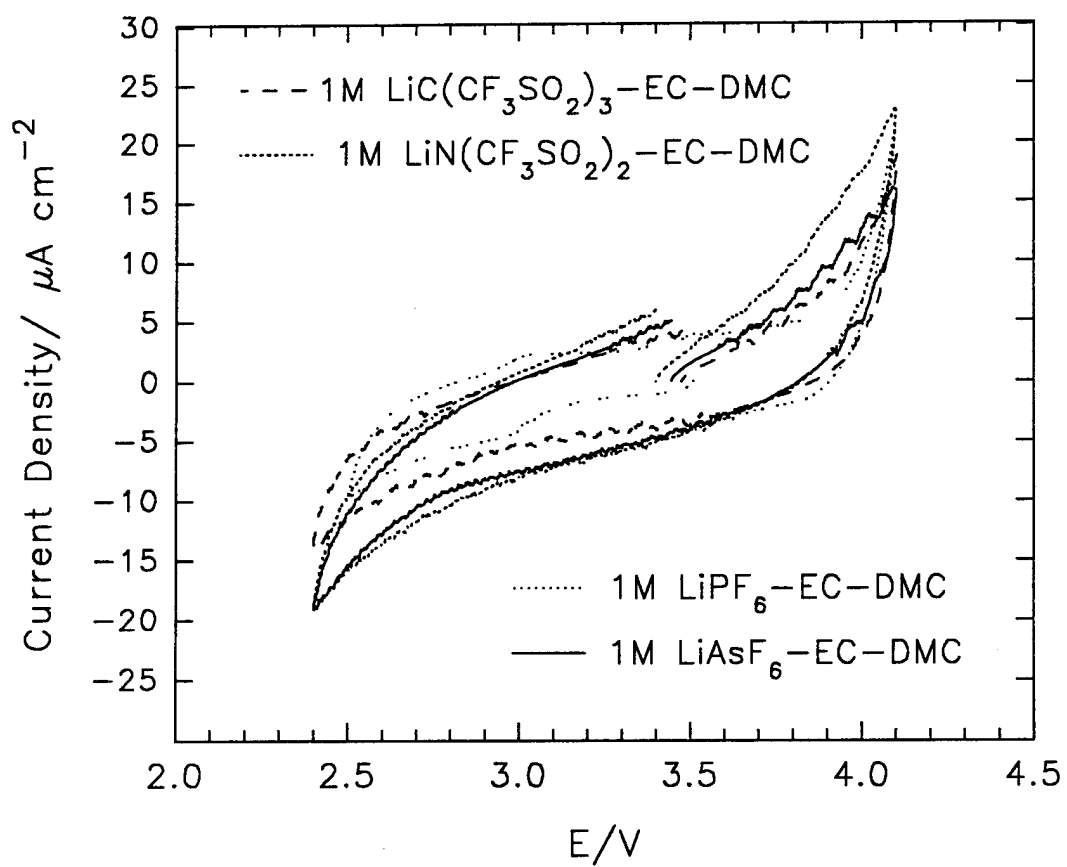


Figure 3. Single sweep cyclic voltammograms of binary solvent electrolytes on glassy carbon, 5 mV s^{-1} .

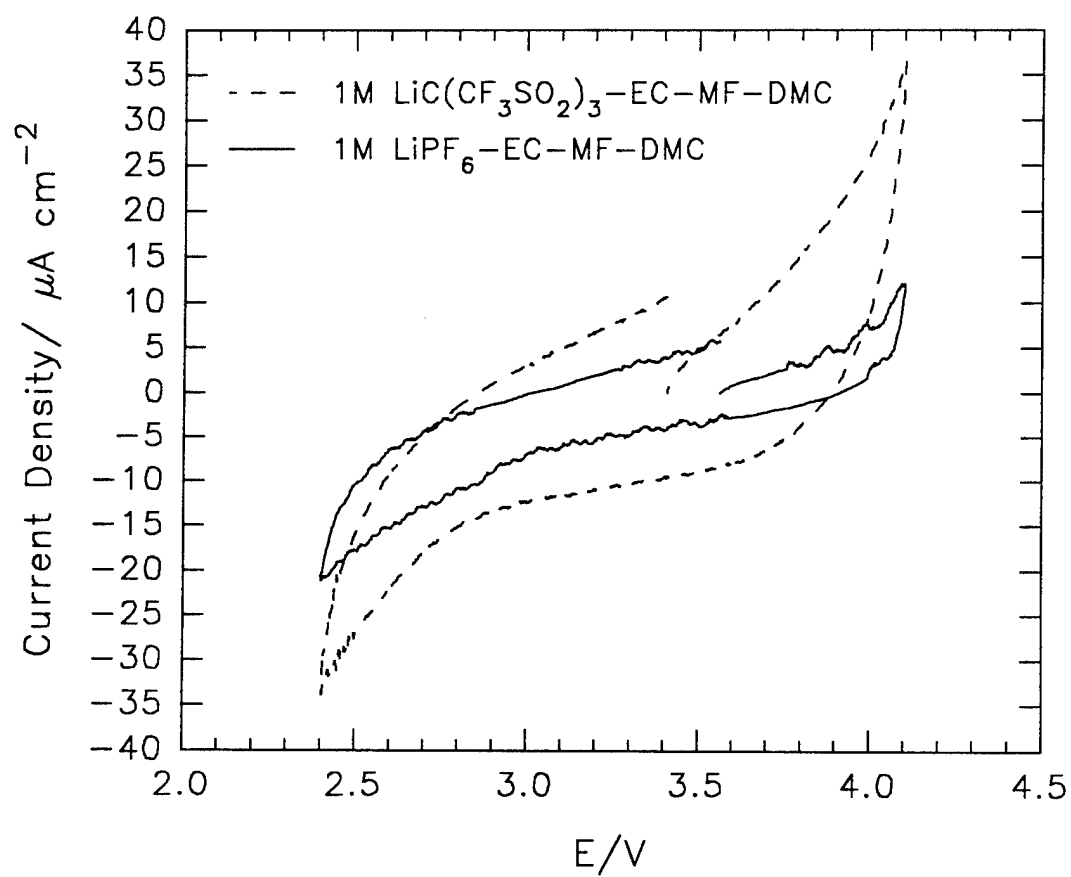


Figure 4. Single sweep cyclic voltammograms of ternary solvent electrolytes on glassy carbon, 5 mV s^{-1} .

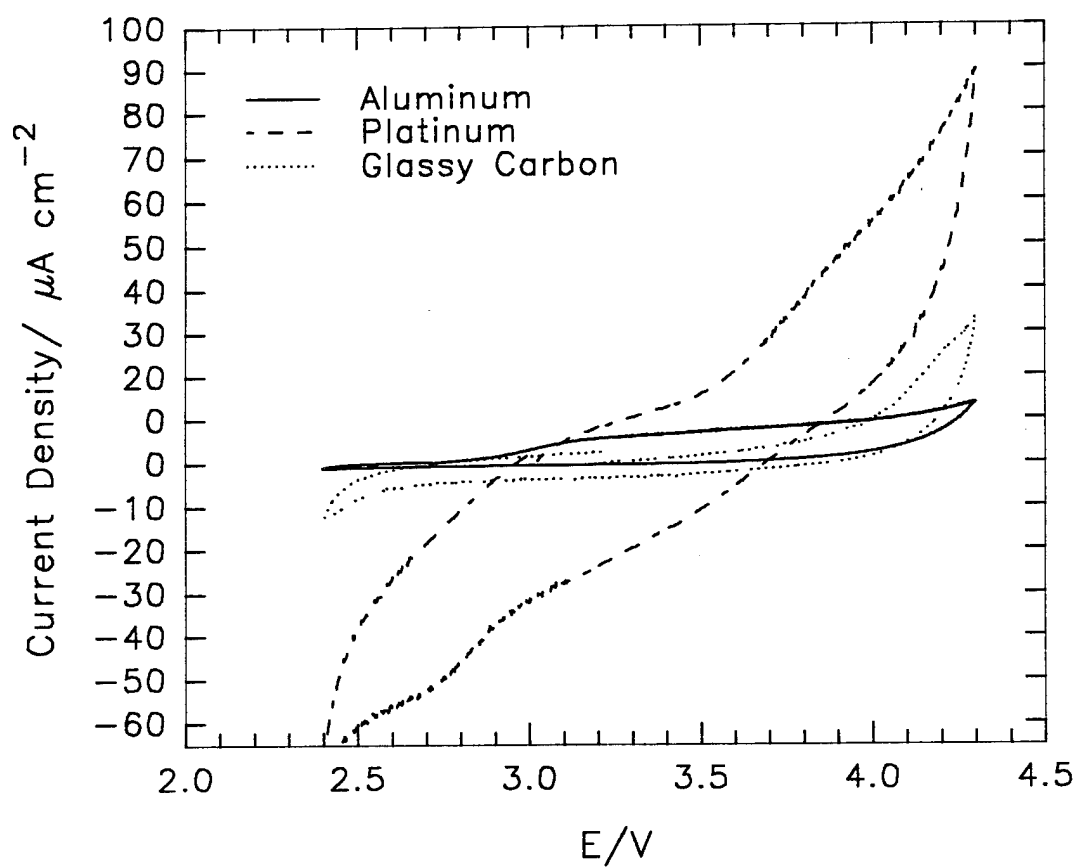


Figure 5. Cyclic voltammograms of $\text{LiN}(\text{CF}_3\text{SO}_2)_2\text{-EC-DMC}$ on aluminum, platinum, and glassy carbon at 5 mV s^{-1} .

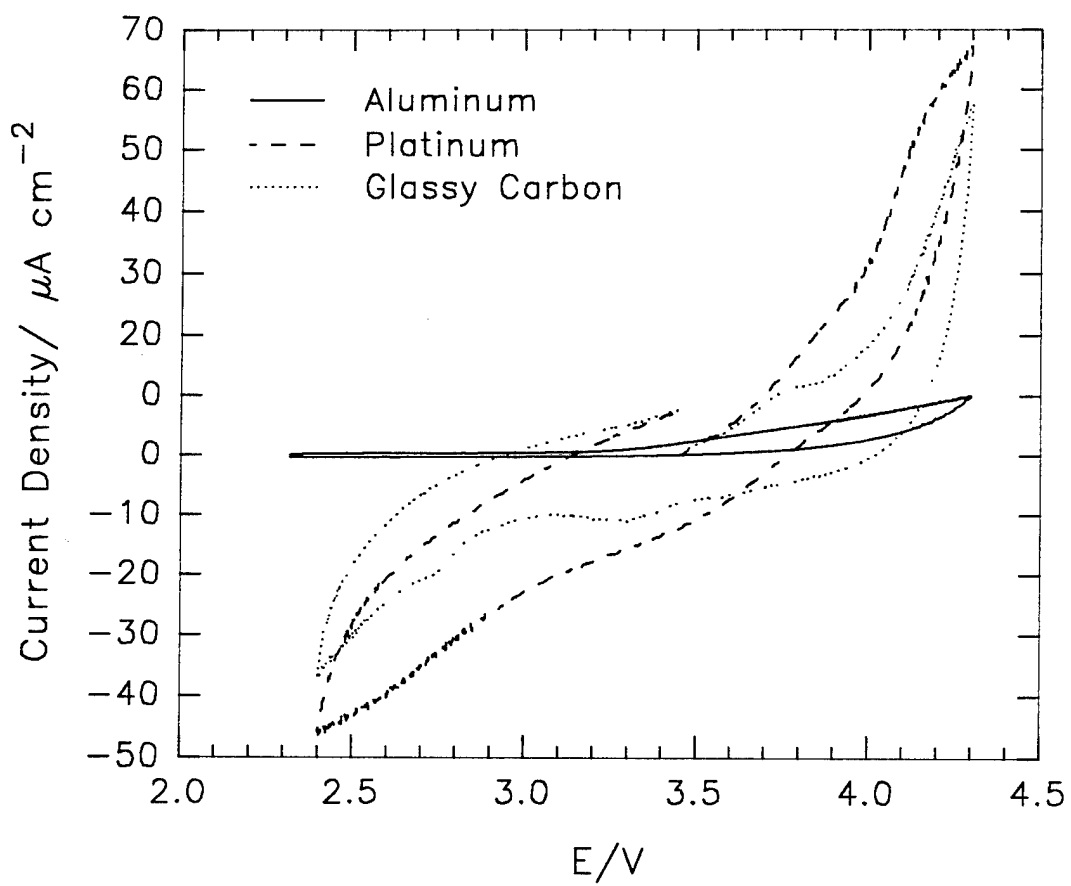


Figure 6. Cyclic voltammograms of $\text{LiC}(\text{CF}_3\text{SO}_2)_3\text{-EC-MF-DMC}$ on aluminum, platinum, and glassy carbon at 5 mV s^{-1} .

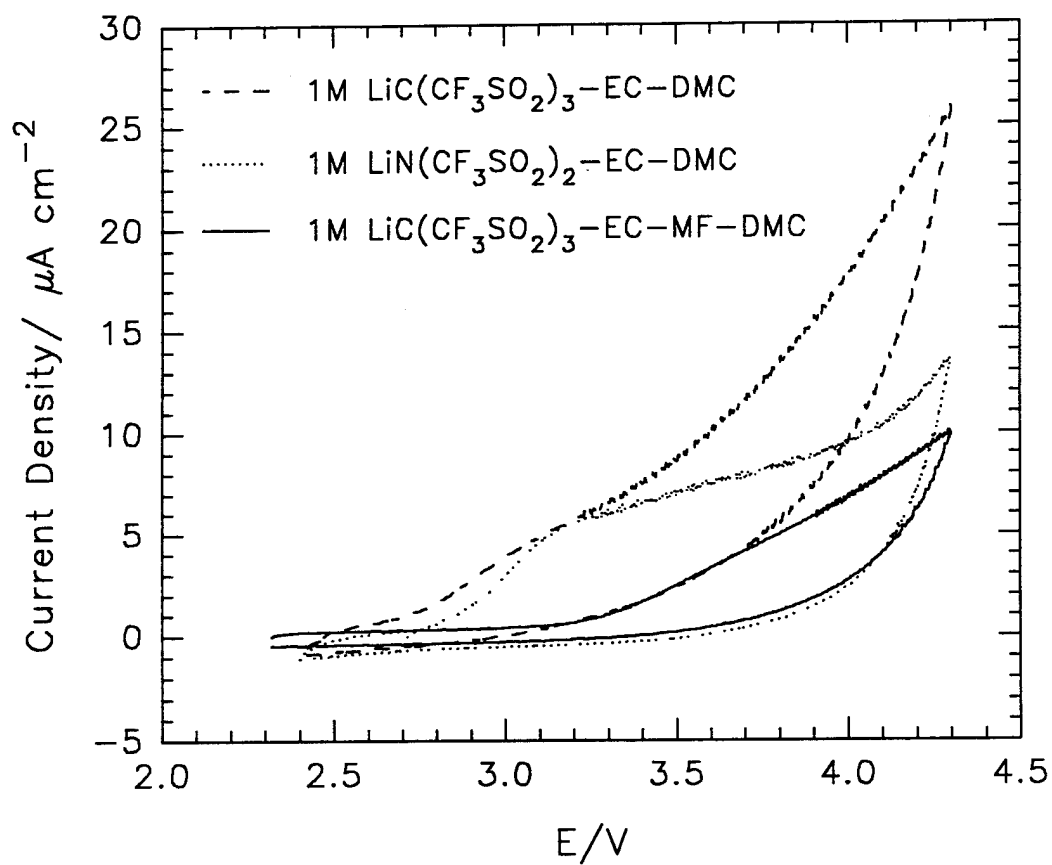


Figure 7. Comparison of methide and imide electrolytes to 4.3 V on an aluminum substrate at 5 mV s^{-1} .

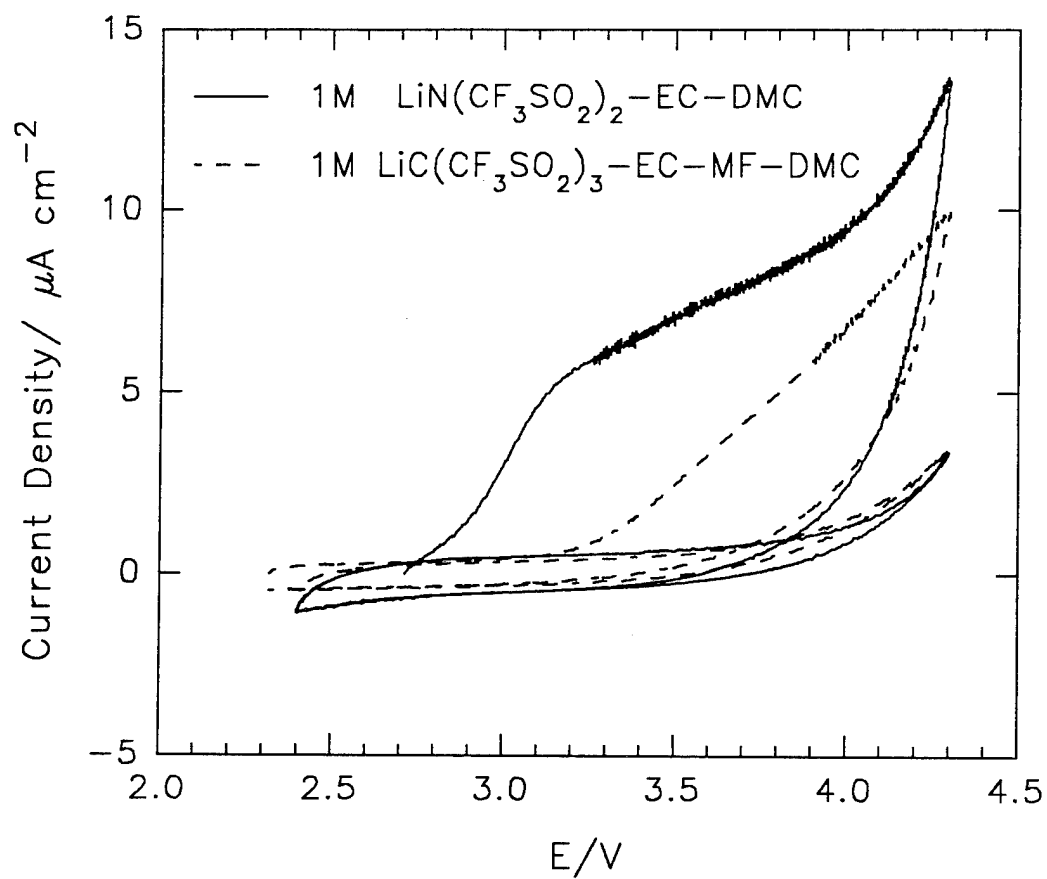


Figure 8. First (upper curves) and fifth (lower curves) cycles on an aluminum substrate to 4.3 V at 5 mV s^{-1} , comparing the methide and imide electrolytes.

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